International Journal of Nanotechnology and Application (IJNA)
ISSN(P): 2277-4777; ISSN(E): 2278-9391
Vol. 6, Issue 2, Apr 2016, 1-6

POLY (ETHERIMIDE)/ POLYPHOSPHAZENE COATED MULTIWALL CARBON NANOTUBES NANOCOMPOSITES

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ABSTRACT

Nanocomposites of Poly (etherimide) (PEI)/ multiwall carbon nanotubes (MWCNTs) modified by Polyphosphazene have been prepared by melt mixing process through co-rotating twin screw extruder. The influence of varied loadings of modified MWCNTs on the mechanical, heat deflection temperature (HDT) and morphological properties of the developed nanocomposites have been investigated. Dispersity of MWCNTs over PEI matrix has been evaluated by scanning electron microscopy (SEM). SEM studies demonstrate that there is uniform and homogeneous dispersion of MWCNTs over the entire PEI matrix. Mechanical tests depict that the nanocomposites have good mechanical properties as compared to virgin composite. The HDT of developed nanocomposites also increases with increase in nanofiller content in PEI matrix. Results also show that optimum HDT value has been achieved at 0.3 wt % loading of MWCNTs.

KEYWORDS: PEI, Polyphosphazene, SEM, Mechanical properties, MWCNTs

Received: Feb 20,2016; **Accepted:** Mar 07, 2016; **Published:** Mar 11, 2016; **Paper Id.:** IJNAAPR20161

Original Article

INTRODUCTION

Polyimides are versatile engineering polymers that have been extensively used in many high-tech and advanced technology applications due to their outstanding thermal stability, mechanical properties etc. PEI resins are amorphous thermoplastics materials that result from the combinations of ether unit and aromatic imides. Ether units provide the chain mobility and good melt flow characteristics to PEI, while the aromatic imides units supply thermal resistance and mechanical properties. Carbon nano tubes (CNTs) have unique and excellent physical properties; therefore they have many structural smart applications **[1-5].** Among them, one of the most outstanding applications of CNTs in the polymer/CNTs nanocomposites.

CNTs are widely used as nanofillers for the fabrications of advanced polymeric composites due to its superior properties. However, uniform and homogeneous dispersion of CNTs in the polymer matrix is the major drawback for the development of high performance nanocomposites. To improve the dispersion of CNTs in polymer matrix, functionalizations of CNTs is very essentials **[6-10]**. The surface modification of CNTs is possible using third phase dispersing agent such as surfactant **[11-13].**

Polyphosphozene is an inorganic polymer having back bone Skill et. Al. (P------N) with various organic pendants groups like amino phenol, diethyl ether. Subsequently high molecular weight of these pendants groups and lack of specific interactions (H-bonding, wander wall forces, charge transfer process etc.) are known to facilitate

reduction of the interfacial energy **[14, 15].**

In the present work multiwall carbon nano tubes (MWCNTs) are modified with Polyphosphazene. Nanocomposites based on polyetherimide (PEI) reinforced with modified MWCNTs have been prepared. Advanced analytical technique has been utilized for the characterization of developed nanocomposites such as SEM, TGA, and FTIR etc.

MATERIALS AND METHODS

Materials

PEI (grade ULTEM 1000) purchased from Sabic innovation plastics (USA) is used as matrix materials. The multiwall carbon nano tubes (MWCNT) and Polyphosphazene (PPH) were synthesised by DMSRDE, Kanpur. MWCNT having a diameter 2-4 nm and length 20-30 micrometer and the aspect ratio 10,000.

Coating of Inorganic Polymer on MWCNT

Took 2 liter tetra hydro furan (THF) and 100gm KOH pass it over 500 gm Alumina to remove the moisture , press sodium in this solution and leave the solution for two days a bluish colour is produced. Distillation of this solution was done and then took 100 ML of this solution in a bottle and adds 100 mg PPH in it. Now took 40 ML of the above solution and add 0.4 gm MWNT in it then reflux it for 6 to 7 hours. Now distillation of the above solution is done at 90^0C then they obtained residues is heated up to 25^0C and then leave it to cool as a result we get PPH coated MWCNT.

Dispersion of Inorganic Polymer Coated MWCNT'$_S$

Before incorporation of PPH coated MWCNT'$_S$ from 0.1 to 0.3 phr in PEI. The nanotubes have been sonicated in 10 ml of toluene for a period of 30 minutes to separate the agglomerated Polyphosphazene coated MWCNT's at in order to induce an efficient dispersion of nanotubes.

Preparation of Organic/Inorganic Polymer Nanocomposites

Before mixing, the organic polymer (PEI) was dried under vacuum at 80^0C for at least 12 hrs. Then after organic polymer and inorganic polymer with nanoclay (Polyphosphozene coated MWCNT) were blended in twin screw extruder (at RPM 145) at temperature processing condition given in Table 1 for PEI blend with inorganic polymer as per formulation Table 2. Testing specimens were prepared using injection moulding machine at temperature 340°C for PEI blend with inorganic polymer / nanoclay.

Table 1: Processing Temperature of Twin Screw Extruder during the Compounding of PEI nanocomposites

Heating Zone	PEI
Zone 1	320 ^{o}C
Zone 2	350 ^{o}C
Zone 3	360 ^{o}C
Zone 4	375 ^{o}C
Zone 5	380 ^{o}C
Die	385 ^{o}C

Table 2: Formulation of the Nanocomposite based on PEI/PPH Coated MWCNT Blend

Sr. No.	PEI (gm)	PPH Coated MWCNT (%)
1.	500	0.1
2.	500	0.3
3.	500	0.5

Testing and Characterisation

Test specimens were prepared by injection moulding for the analysis of Mechanical and thermal properties.

Mechanical & Thermal Properties

Density were measured as per ASTMD 792, Tensile properties were determined using dumbbell shaped specimen as per ASTMD 638, Flexural properties were measured as per ASTMD 790 using universal testing machine (model INSTRON 3382, USA). The thermal properties heat deflection temperature (HDT) was measured at 1.82 MPa as per ASTMD 648. During testing the test atmosphere was maintained at 23+/- 2 deg C and 50+/- 5 RH.

Morphological Studies

Scanning electron microscopy (SEM) techniques was used to analyse the morphological properties of PEI/ Polyphosphozene coated MWCNT. Prior to SEM analysis the tensile strength broken samples were gold coated with the help of gold sputtering unit for avoiding charge effect. SEM studies were done using Carl Zeiss EVO-50*VP low volume scanning electron microscopy.

RESULTS AND DISCUSSIONS

Mechanical and Thermal Properties

The developed nanocomposites demonstrate (Table 3, 4, 5, 6) higher tensile strength, tensile modulus, flexural strength, flexural modulus hardness and impact strength as compared to virgin PEI. The tensile strength and tensile modulus increases upto 0.3 wt % nanofiller loadings because of better dispersion of MWCNTs in PEI matrix and increased cross-linked density that result in excellent reinforcing effect of the nanofiller. Increasing reinforcing efficiency of nanofiller along with the increasing cross linked density increases stiffness and lower the elongation at break [16]. Thus higher tensile strength and tensile modulus of the nanocomposites may due to the orientation of MWCNTs along the direction of applied stress [17]. Tensile strength and modulus of the nanocomposites are 16% and 12% higher than that of the virgin PEI respectively, Tensile properties results are given in Table 4. The flexural strength of the nanocomposites from 163.24 MPa for virgin PEI to 176.34 MPa at 0.3wt % MWCNTs reinforced PEI composites. This is an increase of 8 % in the magnitude of the flexural strength as given in Table 5. The reinforcing effect caused by MWCNTs in the contributions of strong interactions between matrix and nanofillers.

It is also evident from Table 3, that hardness and density of the developed nanocomposites has higher value as compared to virgin PEI. This may be because of excellent dispersion of MWCNTs over the entire polymer surface and also the orientation of MWCNTs in desired direction. Table 5 also depicts a significant enhancement in flexural modulus with the incorporations of MWCNTs in PEI matrix. It is worth noting that higher value of flexural modulus at 0.3wt % of MWVNTs content PEI matrix. The increment has been found to be about 13% in flexural modulus at 0.3wt % of MWCNTs content. These results suggest that modulus enhancement may depends on the dispersion for a given value fraction of filler also depends on the interactions on PEI matrix and MWCNTs.

The variations on the izod impact as functions of MWCNTs content have been tabulated in Table 6. It is evident from the results that the impact strength of nanocomposites are higher than that of virgin PEI. The impact strength of virgin PEI have been found to be 51.0 J/m where as it is 67.0 J/m with the incorporation of 0.3 wt % of MWCNTs in the PEI matrix. The impact strength has increased 31% compared to virgin PEI, this might be attributed to the decreased of ductility of the polymer with the incorporation of MWCNTs. Table 6 depicts the variation of HDT. It is worth noting that virgin PEI has HDT (206 ^{0}C) where as the nanocomposites having 0.3 wt % MWCNTs in PEI, has HDT (215.6 ^{0}C). Thus, we can say that the nanocomposites having 0.3% MWCNTs has HDT 4.6 % more as compared to virgin PEI. This might be attributed to reduction in the chain mobility of polymer matrix by the incorporation of MWCNTs in PEI matrix.

Table 3: Density, Rockwell Hardness of PEI with Inorganic Polymer Coated MWCNT Nanocomposite

Composition		**Density, g/cc (PEI density – 1.26 g/cc)**	**Rockwell Hardness, (M Scale) (PEI Hardness – 108)**
PEI, gm	Filler %	PPH coated MWCNT	
500	0.1	1.28	110
500	0.3	1.28	112
500	0.5	1.29	111

Table 4: Tensile Strength, Elongation and Modulus of PEI with PPH coated MWCNT Nanocomposite

Composition		**Tensile strength, MPa (PEI Tensile Strength – 104.32 MPa)**	**Tensile Elongation, % (PEI Tensile Elongation – 7.11%)**	**Tensile Modulus, MPa (PEI Tensile Modulus – 3406.43 MPa)**
PEI, gm	Filler %	PPH coated MWCNT		
500	0.1	115.23	6.9	3747.34
500	0.3	121.34	6.8	3821.67
500	0.5	117.62	7.0	3786.23

Table 5: Flexural Strength & Modulus of PEI with PPH coated MWCNT Nanocomposite

Composition		**Flexural strength, MPa (PEI Flexural Strength – 163.24 MPa)**	**Flexural Modulus, MPa (PEI Flexural Modulus – 3428.12 MPa)**
PEI, gm	Filler %	PPH coated MWCNT	
500	0.1	172.42	3730.23
500	0.3	176.34	3879.65
500	0.5	174.56	3795.24

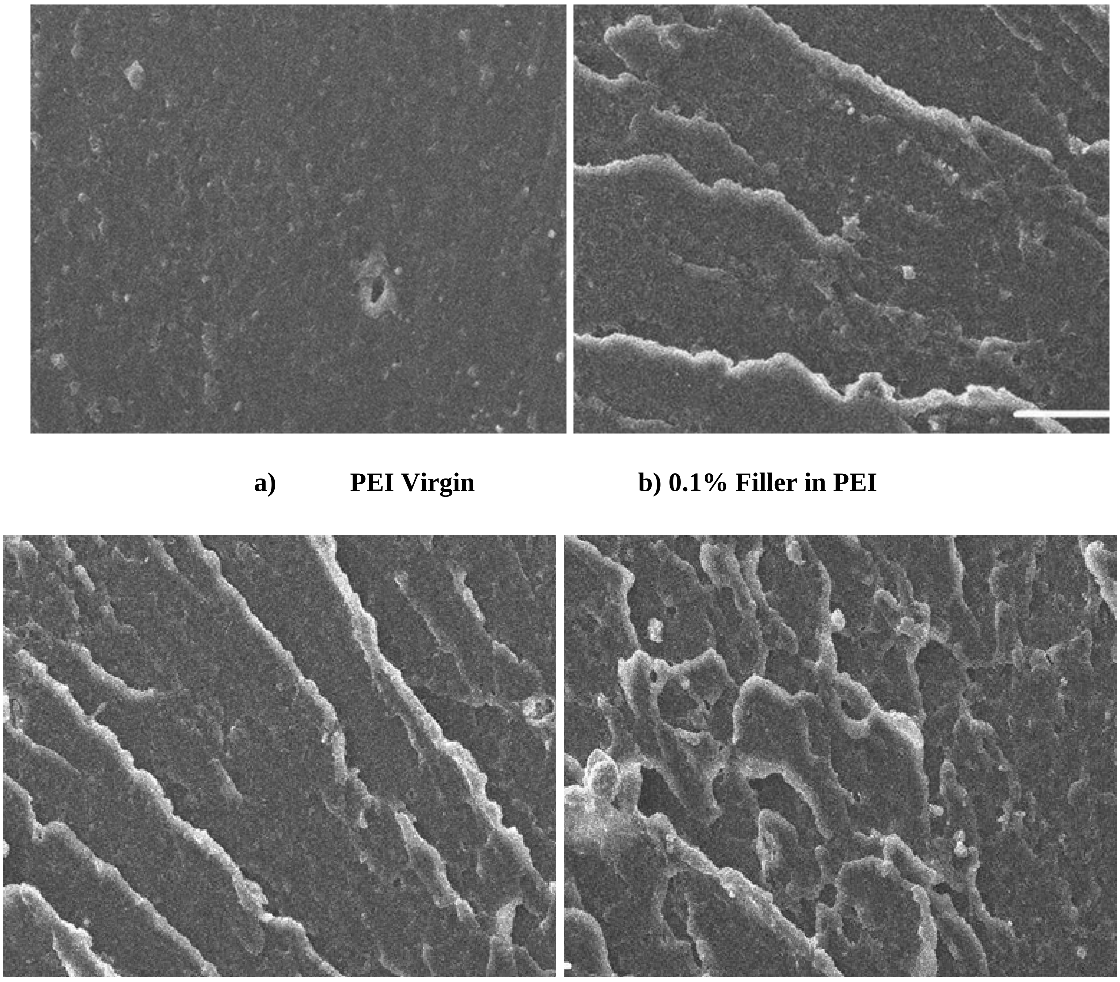
Table 6: Izod Impact & HDT of PEI with PPH coated MWCNT nanocomposite

Composition		**Izod Impact, J/m (PEI Izod Impact – 51.0 J/m)**	**Heat Deflection Temperature (HDT), Deg C (PEI HDT – 206 Deg C)**
PEI, gm	**Filler %**	**PPH coated MWCNT**	
500	0.1	60	208.9
500	0.3	67	215.6
500	0.5	61	210.7

Morphological Properties

Figure 1 (a) shows the SEM micrographs of tensile fractured surface of virgin PEI and the nanocomposites having MWCNTs at various loadings. It is obvious from Figure 1 (a) that for virgin PEI smooth surface can be seen without any

defects or voids. Figure 1 (b) to 1 (d) demonstrate that with the incorporation of MWCNTs there is uniform and homogeneous dispersion of nanofiller and rough surface of MWCNTs gives rise to effective physical interactions and reduces the slippage of MWCNTs. It is believed that with further improvement of MWCNTs clustering via surface modifications through more expensive the mechanical properties further upgraded.



a) PEI Virgin **b) 0.1% Filler in PEI**

c) 0.3% Filler in PEI **d) 0.5% Filler in PEI**

Figure 1: Morphological Properties of PEI/ PPH Coated MWCNT Nanocomposite

CONCLUSIONS

PEI/MWCNTs nanocomposites have been prepared by melt mixing process for better dispersion of MWCNTs in PEI matrix. Almost all the mechanical properties of developed nanocomposites have increased appreciably with the inclusion of MWCNTs in PEI matrix. SEM micrographs depict better dispersion and interfacial adhesion of MWCNTs and PEI matrix.

REFERENCES

1. *Iijima S, Helical microtubes of graphite carbon. Nature 1991: 354 (6348) ; 56-8.*
2. *M. Wang, M. Paramosthy, XJ. Xu. Y, Ren S, LiK, K. Liao, Polymer 44: (2003) 7757-7764.*
3. *L.Shao, Y.P.Bai X. Huang LH, Meng J. Ma J. Appl. Polym. Sci. 113 (2009) 1879-1886.*

4. *J,H,Ko, J.H.Chang, Polymer engg sci. 49 (2009) 2168-2178.*
5. *S. Ion, D. Florian, F. Claudiu, C. Dvidiu, M. Ion. Solid state Sci. 11 (2004) 1793-17-96.*
6. *Hirsch A, Vostrowsky O, Functionalization of carbon nanotubes. Top Curr Chem 2005: 245:193-237.*
7. *Balasubramanian K, Burghard M. chemically functionalized carbon nanotubes. Small 2004: 1:180-92.*
8. *Ganesh Ch., Nayak , R. Rajsekher, CK Das,. Effect of SiC coated MWCNTs on the thermal & mechanical properties of PEI/LCP blend. Compsites : Part A 41 (2010) 1662-1667.*
9. *S. Bose, N. Pramanik, C.K.Das, A. Ranjan, AK Saxena, Synthesis and effect of polyphsphazene on the thermal, mechanical and morphological properties of PEI/LCP blend. Materials and Design 31 (2010) 1148-55.*
10. *S. Kumar, T.Rath, R.N. Mahaling, CS, Reddy, CK Das, K.N.Pandey, R.B. Srivastava, S.B. Yadav. Study on mechanical, morphological and electrical properties of carbon nanofibres in PEI composites. Materials Sci Engg B. 141 (2007) 61-70.*
11. *SD. Bergin, V. Nikolasi, H. Cathcart, M. Lotya, D. Richard, Z.Sum. WJ, Blau JN. Coleman. J. phys chem., C 112 (2008) 972-977.*
12. *P. Angelikopoulosion, A. Gromau, A. Leen, O. Nerushen H, Bock EEB Campbell. J. phys chem., C 114 (2010) 2-9.*
13. *B. Karuse, M. Mende, P. Potschke G. Petzold, Carbon 48 (2010) 2746-2754.*
14. *S. Bose, Mukherjee M. Rath T. Das. C.K. Effect of Polyphosphazene on the properties of blend of nylon 6,6/thermo tropic liquid crystalline polymer. J, Reinf. Plast composites (2009) 28: 157-66.*
15. *Alcock HR, Kugel RL, Valan KJ, Posphonitriclic compounds VI, High molecular weight poly (alkoxy and aryloxyphosphasene) Inorg Chem 1966 : 5: 1709-15.*
16. *Xie W, Gao Z, Liu K, Pan W, Vaia.R, Hinsten D, and sing A, Thermochem Acta 367 (2001) 339.*
17. *Bellueei. F. Camino G. Frach A, and sarra A, Polyme degrade stab. 97 (2007) 425.*